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# (54) MIRROR-SURFACE WAFER, ITS MANUFACTURE AND ITS INSPECTION METHOD (57)Abstract:

PURPOSE: To provide a mirror-surface wafer in which the existence of a latent flaw is not recognized, its manufacturing method and its inspection method.

CONSTITUTION: A mirror-surface wafer which is composed of an Si single crystal is immersed in an etching liquid in which the mixture volume ratio of 50wt.% of hydrofluoric acid, 70wt.% of nitric acid and pure acetic acid is within a range of 1:(1 to 10):(1 to 5); it is etched within a depth range of 0.5 to 15  $\mu$  m from a mirror surface; the mirror surface of the mirror-surface wafer after this treatment is observed by using a differential interference microscope; a finishing polishing operation is performed by using a chemical polishing liquid and a polishing cloth which do not contain any abrasives until no latent flaw is recognized by the observation.

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#### **CLAIMS**

[Claim(s)]

[Claim 1]A mirror surface wafer which consists of an Si (silicon) single crystal 50% of the weight of hydrofluoric acid, it being immersed in 70% of the weight of nitric acid, and an etching reagent which is in the range of 1:(1-10): (1-5) by a mixing volume ratio of each pure acetic acid, and within the limits of 0.5-15 micrometers in the depth from a mirror plane of said mirror surface wafer, [ etch and ] A mirror surface wafer observes a mirror plane of a mirror surface wafer which received this etching process with a differential interference microscope, and existence of a hidden damage is not accepted to be by this observation.

[Claim 2]In the last coloring for manufacturing a mirror surface wafer which consists of Si single crystals, Said mirror surface wafer 50% of the weight of hydrofluoric acid, 70% of the weight of nitric acid, and it being immersed in an etching reagent which is in the range of 1:(1-10): (1-5) by a mixing volume ratio of each pure acetic acid, and within the limits of 0.5-15 micrometers in the depth from a mirror plane of said mirror surface wafer, [ etch and ] A manufacturing method of a mirror surface wafer grinding a mirror surface wafer with chemical grinding liquid and abrasive cloth which do not contain an abradant (abrasive grain) until it observes a mirror plane of a mirror surface wafer which received this etching process with a differential interference microscope and existence of a hidden damage is no longer accepted by this observation.

[Claim 3]A manufacturing method of the mirror surface wafer according to claim 2 which is what is performed after said last coloring passes through one or more steps of polishing processes by chemical grinding liquid and abrasive cloth containing an abradant (abrasive grain).

[Claim 4]A manufacturing method of the mirror surface wafer according to claim 2 which is what is performed after said last coloring passes through a grinding operation by a surface grinding method. [Claim 5]A manufacturing method of the mirror surface wafer according to any one of claims 2 to 4 which is solution containing hydroxide and an ammonia solution of solution or an alkaline metal in which said chemical grinding liquid contains hydroxide of an alkaline metal.

[Claim 6]A manufacturing method of the mirror surface wafer according to any one of claims 2 to 5 which is what is performed until a hidden damage is no longer accepted, when said last coloring observes a mirror plane of said mirror surface wafer with a differential interference microscope.

[Claim 7]A suede type with which said abrasive cloth provided a polyurethane foaming layer on a base fabric, Or are a nonwoven fabric type thing which impregnate foams, and carries out curing treatment, and can obtain polyurethane resin to a base fabric, and weighted solidity of the abrasive cloth surface part, A manufacturing method of the mirror surface wafer according to any one of claims 2 to 6 which is a thing used as the range of the hardness (JIS K-6301) 40-80, the compression ratios (JISL-1096) 2-20, and the elastic compressibility (JIS L-1096) 60-99.

[Claim 8]A mirror surface wafer which consists of Si single crystals 50% of the weight of hydrofluoric acid, 70% of the weight of nitric acid, and it being immersed in an etching reagent which is in the range of 1:(1-10): (1-5) by a mixing volume ratio of each pure acetic acid, and within the limits of 0.5-15 micrometers in the depth from a mirror plane of said mirror surface wafer, [etch and ] An inspection method of a mirror surface wafer which has each process of observing a mirror plane of a mirror surface wafer which received this etching process with a differential interference microscope, and inspecting existence of a hidden damage.

[Translation done.]

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## DETAILED DESCRIPTION

[Detailed Description of the Invention] [0001]

[Industrial Application]This invention relates to a mirror surface wafer (henceforth a wafer) in which it excels in the surface surface smoothness for manufacturing the semiconductor device (henceforth a device) of the degree of high integration like super-\*\*LSI from very large scale integration, and neither a damaged layer nor a hidden damage exists, its manufacturing method, and an inspection method for the same.

[0002]

[Background of the Invention]The Si-single-crystal stick which usually pulled up the wafer which consists of Si single crystals by CZ process or an FZ method, and was manufactured, After carrying out cylinder polish by setting the axis of rotation as the raising medial axis of that stick, cutting vertical constant width regularly to the raising axis continuously and obtaining a disc-like slice, this slice is further manufactured through many processes, such as camfering, wrapping, etching, heat treatment, and mirror polishing. As for these work processes, conditions are set up corresponding to the standard of wafer products. In this case, although another process may be added if needed or processing sequence may be changed, mirror polishing is always performed in a culmination. Thus, the manufactured wafer is washed and dried after that, and it is shipped after undergoing many inspections as a product.

[0003] The above-mentioned mirror polishing had several steps of polishing processes by which the alteration of condition was carried out, such as the secondary polish [ 3rd ], comprised primary polish, and has called polish of the culmination coloring. By the way, the mirror polishing method which makes the present mainstream is called what is called mechanochemical grinding method that compounded the mechanical polish by an abradant (abrasive grain) and abrasive cloth, and the chemical polish by the working liquid of etching nature.

Polish by the principle of one of these is not performed.

This is because the quality and productivity of a wafer with which the conventional device fabrication is presented are based on this method and satisfied.

[0004] For example, when performing mirror polishing by two steps, the alkaline grinding liquid which made the comparatively big and rough colloidal silica abradant whose particle diameter is 30-70 nm suspended is used as the 1st step, With the quick polishing speed (a part for about 1-2-micrometer/) by high polishing pressure, it grinds to near [ predetermined ] the last machining allowance, and the flat surface is obtained. Next, as the 2nd step, a pressure is lowered and a short time for 1 to 10 minutes and soft polish of around 1 micrometer are performed as coloring at the same time it changes an abradant to a thing with a fine particle diameter of 10-30 nm. [0005]Or there is also a method of changing gradually the mechanical conditions of abrasive cloth and polishing work, keeping almost constant the monograph affair of an abradant and

[0006] It dries through washing by a predetermined method, and the wafer which ended coloring undergoes various inspections as wafer products. Contaminant measurement [ like analysis of the outside which mainly inspects the state of a wafer surface portion, and a surface contamination impurity, such as existence of the existence of the appearance defect by the optical means outside the electrical property or particle, display flatness of a mirror surface

part, measurement of surface roughness, ] whose quality inspection as wafer products is is a center.

It does not succeed in the inspection to the inside of a wafer which poses a problem on device fabrication especially the crystal defect which exists near the surface, or a thing like processing distortion selectively.

## [0007]

[Problem(s) to be Solved by the Invention]However, the high integration of a device is becoming severe so that naturally [ the quality requirements to the wafer products which are the raw material ], while it advances without the place at which it stops and goes into the generation of super-\*\*LSI from very large scale integration. That is, although the minimum pattern dimension of very large scale integration is based on 1 micrometers, the 0.25-micrometer lead is taken in super-\*\*LSI, and it is required according to advance of this pattern minuteness making that the mirror planes of wafer products should be more advanced display flatness and advanced crystal quality.

[0008] Although the main part of this crystal quality is determined at the time of raising of a single crystal rod, big influence is received also by the work process for future wafer manufactures. The influence of the 1st is generating of the surface or an internal defect resulting from the impurity contamination which the crystal covered by the work process, and the influence of the 2nd is the existence of a detailed damaged layer and a hidden damage which it is formed mainly in a mechanical work process, and moreover could not be removed by

careful processing either.

[0009] Since the influence of the 1st appears directly in the process in which compound with the original quality of a crystal and a device is manufactured, the break through and measure may be set not only to a wafer supplier but to the user side, and are studied. Namely, the oxidation induction stacking fault (Oxidation InducedStacking Faults: henceforth OSF) formed in the surface part and inside of the wafer expected to be based on the quality or said influence of the 1st of original of a crystal or other minute faults, By throwing into relief in the form which is visible to eyes by the surface treatment by SEKOETCHINGU liquid or a JIRUTORU etching reagent, it can inspect under a microscope. In this case, although the crystal defect of a wafer itself which exists in the surface very much will be easily removed by these etching-reagent processings, in the process in which a wafer is etched from the surface, since the etch rate of the defective part under crystal differs from the etch rate of other non-defective parts, the unevenness which is a wavelength grade of light is observed as a pit or a hillock. [0010]On the other hand, the damaged layer formed in the inside of a wafer by the influence of the 2nd not only has an adverse effect on the electrical property of a substrate crystal, but is a heat treatment process of device manufacturing, it becomes a source of release of a rearrangement or particle, and when extreme, it also becomes a cause of a wafer fracture. Therefore, as for the manufacturing process of wafer products, a process is devised so that this damaged layer may be removed thoroughly, and that inspection is conducted by the thing adapting an electron microscope or X-rays, observation by the optical microscope of the sample body by slanting grinding method, etc.

[0011]However, about existence of this 2nd influence portion and a damaged layer. Although there is an opinion that itself becomes an OSF source of release, the actual condition of it is rare to be taken up as a technical problem which was hard to be referred to as still being solved fully, included into the 1st above influence portion, was processed concurrently, and became

independent.

[0012] The result in which artificers examined the generation cause of the crystal defect in a wafer with various inspection methods paying attention to this problem, The origin of this defect not necessarily only not only in the original quality of a crystal, and the thing to depend on said influence of the 1st, Knowledge that existence of the 2nd influence portion that was not able to be discovered in the inspection method of the old damaged layer, i.e., a potential damaged layer, and a hidden damage (this is called hidden damage below.) is also involving was acquired. [0013] Then, artificers came to complete the sharp inspection method which surpasses a

conventional method, and a mirror surface wafer from which a hidden damage etc. are not detected by the inspection method, either and a manufacturing method for the same, as a result of studying wholeheartedly the inspection method of such a hidden damage, and the method for losing the hidden damage.

[0014]

[Means for Solving the Problem] That is, this invention considers the following inventions as main composition. This invention a mirror surface wafer which consists of an Si (silicon) single crystal 50% of the weight of hydrofluoric acid, it being immersed in 70% of the weight of nitric acid, and an etching reagent which is in the range of 1:(1–10): (1–5) by a mixing volume ratio of each pure acetic acid, and within the limits of 0.5–15 micrometers in the depth from a mirror plane of said mirror surface wafer, [ etch and ] A mirror plane of a mirror surface wafer which received this etching process is observed with a differential interference microscope, and a mirror surface wafer existence of a hidden damage is not accepted to be by this observation is provided.

[0015]In the last coloring for this invention to manufacture a mirror surface wafer which consists of Si single crystals, Said mirror surface wafer 50% of the weight of hydrofluoric acid, 70% of the weight of nitric acid, and it being immersed in an etching reagent which is in the range of 1:(1–10): (1–5) by a mixing volume ratio of each pure acetic acid, and within the limits of 0.5–15 micrometers in the depth from a mirror plane of said mirror surface wafer, [ etch and ] A mirror plane of a mirror surface wafer which received this etching process with a differential interference microscope. A manufacturing method of a mirror surface wafer grinding a mirror surface wafer with chemical grinding liquid and abrasive cloth which do not contain an abradant (abrasive grain) is provided until it observes and existence of a hidden damage is no longer accepted by this observation.

[0016] Furthermore, this invention a mirror surface wafer which consists of Si single crystals 50% of the weight of hydrofluoric acid, it being immersed in 70% of the weight of nitric acid, and an etching reagent which is in the range of 1:(1-10): (1-5) by a mixing volume ratio of each pure acetic acid, and the range of 0.5-15 micrometers in the depth from the wafer surface, [ etch and ] An inspection method which has each process of observing a mirror surface part of a wafer which received this etching process with a differential interference microscope is

provided. [0017] Hereafter, this invention is explained. In a process of manufacturing a device, a wafer often receives heat treatment of around 800-1200 \*\* from a wafer manufactured by a predetermined method. If an impurity and a defect which exist during Si crystal in that case deposit near the surface of a wafer, a crystal defect and a rearrangement will be generated by making it into a core, and it will have an adverse effect to the characteristic of a device. [0018]OSF generated in thermal oxidation processing in a process of manufacturing a device from a wafer is observed especially in this crystal defect. Since thermal oxidation processing is a process indispensable to device fabrication, an inspection for predicting existence of OSF generating to wafer products is conducted. After the method removes an oxide film by fluoric acid after carrying out heat-treatment of about 1 hour for a wafer at 1100-1200 \*\* in an oxygen containing atmosphere, and it processes the mirror surface part with SEKOETCHINGU liquid or a JIRUTCIRU etching reagent, it is observed with the usual optical microscope. 100191Although it is supposed that there are a thing resulting from an impurity, a defect, or quality of a surface contaminant which exists in an inside of a crystal, and a thing resulting from damage at the time of mechanical polishing generating of this OSF, it is difficult generating to distinguish that origin. However, as mentioned above, about the present wafer products, each process is constructed so that damage at the time of such processing may be removed thoroughly.

[0020]However, artificers had a question about whether damage at the time of processing of the present wafer products is removed thoroughly, and they examined how to inspect the surface, performing processing by various etching reagents to a mirror surface part of a wafer manufactured by the conventional mirror-polishing method.

[0021]As a result, although not detected depending on other etching reagents, when observing a

wafer mirror surface part which carried out the surface treatment in a certain condition range with a mixed acid etching reagent of use by this invention with a differential interference microscope, \*\*\*\* of the shape of a scratch unobservable with a usual optical microscope and an optical surface roughness meter was able to be observed.

[0022] This \*\*\*\* is measured with a value of about 10-100 nm, when measuring that surface roughness with a surface roughness plan (WYKO TOPO 3D), while the mixed acid etching reagent performs etching up to about 15 micrometers in the depth from the original wafer mirror plane at 0 micrometers or more. However, if etched depth of not less than about 15 micrometers is reached, the granularity will change to a wave of the shape of a big wave, and \*\*\*\* observed in the original mirror plane will become indistinct.

[0023]On the other hand, initial observation images of CW side (field which has not received mirror surface finish) of an opposite hand differ from a wafer mirror plane clearly with a case of the above-mentioned mirror plane, and, moreover, the observation images hardly change in process of a mixed acid etching process. Therefore, it produced clearly on the occasion of mechanochemical polishing, and \*\*\*\* of the shape of said scratch observed by this wafer mirror plane side was conjectured that it is unobservable and to be \*\*\*\* (hidden damage) which exists in the depth of 10 nm or less of numbers from a surface layer of a mirror plane by the usual inspection method.

[0024]As a result of an artificer's etc. examining many things about a cause which this hidden damage generates, in a mirror-polishing process, reasoning that it will be what is produced when a Si surface is scratched by abradant (abrasive grain) contained in abrasive soap was reached. [0025]Then, when coloring was performed having used only chemical etching nature grinding liquid and abrasive cloth which do not contain an abradant, and observing a generation state of a hidden damage with a hidden damage inspection method of this invention, it was checked that wafer manufacture from which a hidden damage is not detected by said method is possible. And it was checked that generating of OSF is controlled compared with a wafer of the former [ wafer / this ], and a technical problem of this invention was solved.

[Function] Although the abrasive soap and abrasive cloth which were suspended to lye by using several 10-nm colloidal silica as an abrasive grain with mean particle diameter are used together in the mechanochemical grinding method in the conventional mirror-polishing process, even when [ which will generally produce generation of heat, agglutination, etc. in a friction surface if an object is printed ] it comes out and wipes in a soft polishing cloth surface, When the abrasive grain adhering to it \*\* the hydration film which exists in a polished surface, it is broken through, and it is thought that abrasive grain size, and a mechanical damage and a scratch with the almost new level were generated on the mirror plane. However, it is thought that such detailed damage (hidden damage) was not detected depending on the conventional inspection method. [0027] By the way, that surface can oxidize by operation of an oxidizer first, and the model of repeating the reaction which hydrofluoric acid acts and dissolves in this oxide can explain etching of Si. For example, when SEKOETCHINGU liquid and the JIRUTORU etching reagent which are well known as an etching reagent are taken for an example, a powerful oxidizer is blended with K2Cr2O7 at the former, and is blended with CrO3 by each at the latter, and the emainder comprises hydrofluoric acid and water. Although the former is suitable for etching of a <100> direction crystal and the latter is suitable for etching of a <111> direction crystal, it may be used without distinguishing especially.

[0028]On the other hand, an oxidizer is nitric acid and the etching reagent of adoption by this invention adds acetic acid to this outside hydrofluoric acid. Therefore, the point of difference of the etching operation which appears when various kinds of etching reagents are made to act on the mirror plane of the common wafer which has not received any pretreatment (for example, thermal oxidation processing), What will be been a thing based on the difference of the method of oxide film formation over a wafer surface and the method of the dissolution of the oxide film is imagined easily.

[0029]That is, in the case of the conventional etching reagent, an oxide film is first formed in a macroscopic form, and it is possible to oxidize at once and to conceal such a detailed damaged

part. On the other hand, with the etching reagent of adoption, by this invention. Since in addition to the character as an original oxidizer of nitric acid a kind of buffer effect by the concentration and acetic acid acts and oxide film formation and its dissolution of a form very more micro than the layer part of a mirror plane are repeated, Also in advance of the etching process of an about [ 0.5–15 micrometers ], the crack with such the detailed surface is continued in the depth from a mirror plane, without being erased, and it is thought that the image is observed by a differential interference microscope.

[0030]It is because it is one of the purpose of a latter device observing detailed unevenness of a mirror surface part that this image is not observed with the usual optical microscope, but is observed only in the view of a specific condition with a differential interference microscope, therefore the polarization device is used.

[0031]As a result of adding the coloring process only by the grinding liquid which eliminated the abrasive grain in abrasive soap like this invention from reasoning that generating of such a hidden damage is a thing based on an abradant (abrasive grain), the hidden damage in question disappeared. And in such a wafer, it was checked that a processing crack decreases compared with the case where generating of OSF believed to be the cause is the conventional wafer. [0032]

[Example]

Example 1 and Example 2 — it examined about the inspection method of the hidden damage of a wafer mirror plane first. Here, as shown in Table 1, the wafer which has eight kinds of various electrical properties of No.1 — 8 was prepared. These wafers are manufactured from the Sisingle-crystal stick by a CZ process, are 125 mm in diameter, and are wafers by which mirror surface finish was carried out with the usual mechanochemical grinding method. SEKOETCHINGU liquid (comparative example 1) and a JIRUTORU etching reagent (comparative example 2) were prepared as the mixed acid etching reagent (examples 1 and 2) of a presentation as shown in Table 2, and a comparative example. And about the wafer which uses these four sorts of etching reagents, and has each above-mentioned electrical property. The mirror surface part which etched on the conditions that the machining allowance from a mirror plane is set to 3 micrometers, and was etched after that was observed for the 50 time magnification by the usual optical microscope and a differential interference microscope (NIDECK IM-8A).

[0033]As a result, in the usual optical microscope, a substantial change of the image was observed about no cases. On the other hand, when it observed with a differential interference microscope, in processing by the etching reagent of Example 1 and Example 2, [ various / wafer ], scratch-like \*\*\*\* was observed after etching and change was accepted. However, by processing by the etching reagent of the comparative example 1 and the comparative example 2, a substantial change of the mirror plane image was accepted [ in / no / the wafer ] by the surface state before etching, and the surface state after etching. These audit observations are shown in Table 3.

Table 3 ----- Sample wafer Example 1 example 2 comparative-example 1 Comparative example 2 -----No.1 changeful Changeful With no change . with no change No.2 -- changeful Changeful . with no change With no change No.3 -- changeful . It is changeful. With no change With no change No.4. It is changeful. Changeful With no change With no change No.5 Changeful Changeful With no change With no change No.6 Changeful Changeful With no change With no change No.7 Changeful Changeful With no change With no change No.8 Changeful Changeful With no change With no change ----- [0037] having used it in example 3 Example 1 -- the same -- the influence by etching cost was examined about four sorts of wafers which have the various resistivity of P type and N type by crystal orientation <100>. That is, to these wafers, the same mixed acid as Example 1 was used, it set up so that the machining allowance might be set to 0.5, 3.0, 6.0 and 10, and 15 or 20 micrometers, and it etched, respectively, and change of the scratch image in the process was observed. [0038]As a result, although the scratch image was observed in all those cases, the image of a scratch line fades with deepening of etching cost. The place which incidentally measured the surface roughness with the surface roughness plan (WIKO TOPO 3D), By 10-micrometer etching, unevenness of 50-100 nm and an etching surface was expanded to 10-50 nm by 3micrometer etching, and that whose granularity of the original mirror plane was a small wave of 10 nm or less changed to the wave of the big shape of a 100-500-nm wave by 20-micrometer etching. Therefore, if such an expansion phenomenon of surface roughness and the futility on the work by taking many etching cost are taken into consideration, it will be thought that the range of 0.5-15 micrometers is suitable for etching cost. [0039] About the wafer manufactured by the example 4 usual mechanochemical grinding method, observation by the above-mentioned inspection method was performed, existence of a hidden damage was checked and the examination for removing the hidden damage was done. First, with the P type and N type of crystal orientation <100>, from the Si-single-crystal stick of the resistivity 14 - 18-ohmcm, the wafer as a conventional article with a diameter of 125 mm in which mirror-polishing processing was carried out by the predetermined process was manufactured, and this was made into the comparative example 3. [0040] The conditions of mirror surface finish were carried out as follows. The thing of the range of the hardness 80-60, the compression ratios 5-15, and the elastic compressibility 70-80 which use primary order [ 2nd ] as a velour type and to which it uses 3rd order as a suede type was used for abrasive cloth. By colloidal silica, the abradant was divided into the conditions of the three-stage about the solution of the NaOH concentration 1 - 0.3wt.% as the mean particle diameter of 30-15 nm, and lye, and performed polish for about 10 minutes about each stage. The wafer used six batches of one grinder as one lot, and manufactured wafer three lots each of P type and N type. [0041]When the etching process which reaches the 5-micrometer depth with the mixed acid of use in Example 2 was performed and the mirror surface part was inspected with the differential interference microscope about N type wafer one lot / [ the P type of this comparative example 3, and 1 each, and 6 sheet, the scratch-like hidden damage was detected about the wafer of total. [0042]Next, mirror polishing by the following conditions was added as Example 4 to two lots each / 12 sheet of P type and N type manufactured by the method of the above-mentioned comparative example 3. That is, hardness used the thing of 80, the compression ratio 15, and the elastic compressibility 90 with the suede type of polyurethane, and abrasive cloth performed polish for about 30 minutes with the NaOH aqueous solution of 0.2wt.% which does not contain an abradant. Then, after performing the etching process which reaches the 5-micrometer depth

with the mixed acid of use in Example 2 about each a lot/6 wafers, the existence of the scratch of the surface was inspected with the differential interference microscope. As a result, it was

[0043]Six wafers each of P type and N type left behind by the example 5 aforementioned comparative example 3, After performing each heat treatment for 100 minutes at 1100 \*\* into an oxygen containing atmosphere about six wafers of P type and N type left behind in Example

observed that the scratch image is mostly extinguished in every wafer.

4, OSF which processed with SEKOETCHINGU liquid and deposited in the mirror surface part under 400 times as many microscopes was measured. Measurement of this OSF is the average value measured from the five-point view of the wafer mirror surface part. Table 4 shows the value which averaged the OSF laboratory data of six wafers each further. The result of Table 4 shows that generating of OSF is also controlled about the wafer from which the hidden damage was removed.  [0044]
Table 4 Conductivity type Resistivity (omegacm)
Wafer of the wafer example 4 of the comparative example 3
piece/cm <sup>2</sup> P type 15.0-18.0 27 piece/cm <sup>2</sup> 6 piece/cm <sup>2</sup> N type 14.5-17.5 33 piece/cm <sup>2</sup> 7 piece/cm <sup>2</sup> [0045] [Effect of the Invention] Also with the sharp inspection method in this invention, a mirror surface wafer in which a hidden damage etc. are not detected, and a manufacturing method for the same can be provided. By this invention, the sharp inspection method which detects the existence of the hidden damage of a mirror surface wafer can be provided. According to this invention, generating of OSF becomes possible [ providing the silicon wafer which has little advanced crystal quality].
[Translation done.]